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FABRICATION OF GRANULE AND PELLET HEAT SOURCES FROM OXALATE-BASED $^{238}PuO_2$

by

D. F. Bickford and D. T. Rankin

Savannah River Laboratory
E. I. du Pont de Nemours and Co.
Aiken, South Carolina 29801

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ABSTRACT

Suitable fuel forms for radioisotopic thermoelectric generators are granules of high internal density (>95% of theoretical) or geometric shapes (80 to 90% dense) such as pellets or spheres. Both forms can be made from calcined ^{238}Pu(III) oxalate. The conditions for processing PuO_2 are controlled during fuel form fabrication to ensure pellet integrity; to control density, grain size, and porosity distribution; and to minimize the fraction of potentially respirable fines.

The competing phenomena of expansion caused by radiation damage (including helium generation from radioactive decay of plutonium) and shrinkage caused by sintering must be controlled to assure dimensional stability. The variation of microstructure and related physical properties with process parameters is discussed.

+ The information contained in this article was developed during the course of work under Contract No. AT(07-2)-1 with the U. S. Energy Research and Development Administration.

INTRODUCTION

Plutonium-238 has been produced at the Savannah River Plant (SRP) since 1961. The high heat generating rate (~0.4 watts/g as oxides of the plutonium metal, of which 80 wt % is ^{238}Pu and the remaining 20 wt % is ^{239}Pu and heavier plutonium isotopes), long half-life (87.4 years), and relatively low amount of penetrating radiation make plutonium an ideal source of heat for radioisotopically powered, thermoelectric generating systems. Several fuel forms, including ^{238}Pu metal, $^{238}PuO_2$ microspheres, and $^{238}PuO_2$-Mo cermet disks, have been used to power systems in the Transit, Pioneer, Apollo, and Viking missions [1]. Spherical forms of $^{238}PuO_2$ with a thermal power rating of 100 watts each will be used as heat sources in the higher powered, high-operating temperature system, the Multi Hundred Watt Radioisotopic Thermoelectric Generator (MHW-RTG) required for the Lincoln Experimental Satellites and for missions to the outer planets. Granular forms of $^{238}PuO_2$ are planned for the heat source in the Milliwatt RTG which is being developed for several Department of Defense programs.

At the request of the United States Energy Research and Development Administration (ERDA), a production facility to fabricate $^{238}PuO_2$ fuel forms is being constructed at SRP. An oxalate-based process [2] developed at Los Alamos Scientific Laboratory (LASL) is being adapted for use in this new SRP production facility. Calcined Pu(III) oxalate powder as presently produced at SRP will be processed directly into the desired fuel forms using powder ceramic processes.

This paper describes results of the on-going process development studies at the Savannah River Laboratory (SRL). Structural

determination of the feed powder, intermediates, and final products using optical and scanning electron microscopy is the principal mode of assuring product uniformity. Particle size, density, and chemical purity measurements complement the microscopy results.

PRODUCT REQUIREMENTS

The fuel form or shape, dimensions, and powder density are specified for all radioisotopic fuel systems. In addition, each system undergoes a series of safety tests before the system is qualified for use. These safety tests are designed to assure safe, continuous operation, and containment of the fuel during unusual or accident conditions. The mechanical and thermal properties of the fuel which are primarily controlled by the microstructure determine the response of the fuel to extreme conditions. The properties of primary concern are dimensional stability, strength, thermal shock resistance, and the amount of potentially respirable fines.

Two competing phenomena, radiation damage and sintering behavior, must be balanced to assure dimensional stability. The first phenomenon, radiation damage, tends to cause the heat sources to swell. The primary source of heat from ^{238}Pu is radioactive decay to ^{234}U. The resulting UO_2 lattice is isomorphous with the PuO_2 lattice and therefore presents minimal problems. The alpha particle, produced during radioactive decay, transforms to helium, which causes lattice expansion by atom displacement and subsequent helium entrapment. At elevated

temperatures, the helium becomes mobile and can coalesce at grain boundaries. The resulting bubbles tend to cause additional swelling of the structure.

The second phenomena is sintering behavior. Through mass transport, the microstructure seeks a minimum free energy by minimizing total surface free energy and interfacial free energy and by reducing the potential energy of pressure-volume work stored in closed pores filled with gas. The overall effect of sintering, in this context, is the elimination of pores, thereby producing a coarsening and shrinking of the fuel form.

High strength is desired to prevent fracture caused by stresses arising from launch vibrations and landing impact. The best microstructure for high strength would be a uniform, finely divided porosity with small grain size. High strength may also prevent fracture arising from thermal stresses during reentry or accidental fire. However, if the thermal stresses are so severe that fracture cannot be avoided, a non-uniform microstructure with large pores and large grains would minimize the damage [3].

Safety of any fuel form is based on a minimum of respirable fines being formed under any condition, normal or accidental. The primary biological hazard of $^{238}PuO_2$ is physical uptake by respiration or ingestion. Production of heat sources with good strength and thermal shock resistance also requires minimizing the biological hazard by reducing the probability of breech of containment. In the unlikely event that containment is breeched,

a minimum amount of potentially transportable fines is desirable. Therefore, if fracture or impact cannot be avoided, fuel microstructures which yield a minimum of fines may be more important than high strength.

GENERATOR SYSTEMS

Multi-Hundred Watt Radioisotopic Thermoelectric Generator (MHW-RTG)

Production of MHW fuel is scheduled to begin at SRP in July 1977. As presently specified, the fuel form will be pure plutonium dioxide (PPO) spheres, which are 1.465 ±0.015 in. in diameter and produce 100 ±2 watts (thermal). The specifications are met by hot-pressing ∿250 g of $^{238}PuO_2$ feed into a PPO sphere of about 82.5% of theoretical density (Figure 1). The MHW spheres are then encapsulated in preformed hemispheres of iridium metal by TIG welding.

The operating temperature of the PPO sphere-iridium interface is calculated to be about 1350°C, a temperature where dimensional stability can be a problem. A specific $^{238}PuO_2$ microstructure of 80 to 85% theoretical density (TD) has been demonstrated to remain stable for year-long periods at temperatures up to 1440°C. This stable microstructure was produced by hot pressing granules which had been sized to less than 297 μm and heat treated at 1050 to 1200°C. This stable microstructure is characterized by coarse porosity, well-defined remnants of the granules, and very small "neck" areas between granules.

An unstable microstructure is produced where the fuel form is fabricated by hot-pressing ball-milled powder directly to about 85% TD without granulation. The uniform microstructure that is formed continues to sinter at fuel operating temperatures (1350°C) and reentry temperatures (to 1800°C); networks of cracks also open on the surface.

High mechanical strength in the MHW fuel form has been deemed desirable as a result of high-speed impact tests. During impact, fuel forms with the highly stable structure described above tend to deform the iridium containment capsule beyond its ductile limit, causing a breach of containment. Spheres fabricated from higher strength thoria and impacted under similar conditions tend to remain intact, with little or no penetration of the containment shell.

Both mechanical strength and resistance to sintering are enhanced by rounded, rather than acicular, porosity. Generally speaking, however, the requirements for high strength and for dimensional stability during sintering are in conflict. Strength is enhanced, but stability is decreased, by increasing the ratio of fine porosity to coarse porosity.

If the satellite fails to make a stable orbit, or if a stable orbit is lost after many years of operation, the resulting frictional heating by the atmosphere during reentry could produce temperature gradients as high as 500°C across the diameter of a fuel sphere. Containment would likely be lost during impact if the thermal shock resulted in a few large fragments with sharp

edges. Asymmetric thermal ramping of the dimensionally stable structure described above produces no apparent damage, while fuel forms fabricated without granularization tend to fracture. This is in accordance with Hasselmans' hypothesis [3] that in situations where thermal shock of a monolith will exceed the tensile strength of the material, it is better to introduce flaws (large pores) deliberately rather than produce a high-strength piece (i.e., one with small pores).

Milliwatt Radioisotopic Thermoelectric-Generator

Process development at SRL of Milliwatt RTG fuel is based on an expected fuel form of dense (>95% TD) $^{238}PuO_2$ granules. These granules are fabricated directly from oxalate-based $^{238}PuO_2$ fuel by powder ceramic processes and then sintered to high density (Figure 1). Approximately 8.8 g of granules and 0.4 g of yttrium chips are vibratory-packed in a tantalum alloy (T-111) capsule to a power density of 2.22 thermal watts/cc. After the capsule is sealed by welding, the yttrium is reacted with the $^{238}PuO_2$ during a subsequent heat treatment to reduce the oxygen partial pressure of the fuel. This reduces the potential of oxygen embrittlement of the tantalum.

The operating temperature of the granule-tantalum interface is less than 800°C; therefore, dimensional stability of the granules should not be a problem. No additional sintering should occur, particularly since the fuel granules are greater than 95% of theoretical density. The helium is essentially trapped in the

lattice at temperatures below 800°C and does not coalesce into bubbles large enough to be seen by an optical microscope.

PROCESS DEVELOPMENT

Multi-Hundred Watt Fuel Form Process

Limitations

Process development studies at SRL to date have been limited to right cylindrical pellets weighing 2 to 5 g because of equipment limitations. Therefore, conclusions of effects of process variables on the microstructure are considered qualitative at this stage. Full-scale tests will be made when experimental facilities are available next summer.

Process Description

The planned flowsheet for the fabrication of MHW spheres (Figure 1) consists of processing $^{238}PuO_2$ in three main forms: as a powder, as granules, and finally as an integral, 100-watt sphere. Typical particle morphology (size and shape) of the calcined $^{238}PuO_2$ powder is shown in Figure 2. The powder consists of laths occurring individually or combined to form agglomerates. The particle size distribution is as shown in Figure 3. The large distribution occurs primarily by the formation of agglomerates of the individual laths. Tap density of this powder is approximately 2 g/cc.

The calcined powder is heated in an atmosphere of pure $^{16}O_2$ gas to replace ^{17}O and ^{18}O to form $^{238}Pu^{16}O_2$. This replacement

reduces the neutron radiation by a factor of 3 to 5. Next, the powder is outgassed to remove entrapped helium and to normalize the powder to a specified thermal history. Then, the dry powder is ball milled to the required particle size.

Granules are fabricated to control the sinterability prior to consolidation into the desired fuel form. The milled powder is first cold-pressed into a pellet. Then, the pellet is broken up by forcing it through a 125 μm sieve to form granules less than 125 μm in size. Next, the granules are heat-treated to reduce their sinterability further and to increase their strength.

The spherical MHW fuel form is fabricated by hot pressing the sized and heat-treated granules. Dimensional control is maintained by controlling the dimensions of the die assembly and the sinterability of the feed. Each fuel form is heat-treated after hot pressing to remove volatile impurities, to adjust the stoichiometry of the as-pressed material from about $PuO_{1.85}$ to $PuO_{2.00}$, and to stabilize dimensions and grain size. A grain size of more than 10 μm is desirable to minimize residual sinterability and to minimize fines should intergranular fracture occur. Next, the $^{238}PuO_2$ fuel form is encapsulated in iridium, tested, and prepared for assembly in the RTG.

Effects of Process Variables on Microstructure

The distribution of the amount and type of porosity in the microstructure of a fuel form is affected by the granule size, temperature of the granule, heat treatment, and the pressure

during hot pressing. Decreasing the granule size, decreasing the temperature of the granule heat treatment, and increasing the pressure during hot pressing result in higher density, and a more uniform distribution of the residual porosity (Figure 4). The final grain size is determined by the temperature and time of the final heat treatment of the fuel form after hot pressing. These process variables and their effects on the microstructure are discussed in more detail below.

Granule Size

Decreasing the maximum granule size from less than 297 μm to less than 125 μm results in a more uniform porosity and better granule-to-granule contact (Figure 5). Use of the milled powder directly without the granulation process virtually eliminates the coarse porosity which is required for dimensional stability. Further, at high fuel operating temperatures, the fuel form becomes dimensionally unstable as the material continues to sinter or densify because the microstructure is made more and more uniform, eventually approaching the condition of having no coarse porosity.

Granule Heat Treatment and Pressure during Hot Pressing

Increasing the pressure during hot pressing reduces the coarse porosity between granules; thus, the stability of the microstructure is reduced. Pellets made from granules heat-treated at 1300°C, however, retain most of the granule-to-granule porosity. The rounding of the granule surfaces and coarsening

of the granule internal structure with increasing heat-treatment temperatures accounts for the difference in pellet microstructure.

Granule Morphology

Scanning electron microscope analysis of granules shows that surface rounding and coarsening of the structures occurs as the granule heat-treatment temperature is increased from 1050 to 1300°C. The granules look quite similar at low magnifications (200X). However, at higher magnifications, especially at 5000X, the effect of increasing the temperature becomes apparent. Granules heated at 1050°C appear fluffy with much surface area. At 1175°C, the granules show some surface rounding and coarsening of structures; at 1300°C, the surfaces are rounded, and the structure is coarsened considerably (Figure 6).

Pellet Microstructure

Differences in microstructure caused by the heat treatment temperature of the granules as well as from the pressure during hot pressing can be seen in the following figures. Low temperatures (<1200°C) and high pressure (5000 psi) reduce the coarse porosity between granules. Pellets pressed at 5000 psi from granules heated at 1300°C and all pellets pressed at 3000 psi had a large amount of coarse porosity (Figure 7). A high ratio of coarse-to-fine porosity stabilizes the microstructure and prevents excessive shrinkage at high temperature by increasing the average diffusion distance for material transport. Thus,

fuel fabricated at the higher pressure of 5000 psi has a lower ratio of coarse-to-fine porosity than fuel fabricated at 3000 psi (Figure 8).

The microstructures of pellets that were hot pressed at 3000 psi from granules heat-treated at 1050, 1175, and 1300°C are similar (Figure 9). All three microstructures have a large amount of coarse granule-to-granule porosity. Because of this coarse porosity, microstructures should be reasonably stable at MHW fuel operating temperatures. However, a difference can be observed in the microstructures of pellets hot pressed at 5000 psi from granules heat-treated at 1050, 1175, and 1300°C. The pellet made from granules heated at 1300°C has considerable coarse granule-to-granule porosity, whereas the other two pellets have little coarse porosity (Figure 10). These latter two microstructures will not be as dimensionally stable at high temperature. Apparently, the surface rounding and coarsening that occurred at 1300°C (Figure 11) reduced the driving force (total surface energy) to enhance densification and provided sufficient strength for the granules to resist fragmentation by the applied pressure during hot pressing.

Another effect of granule heat treatment temperatures can be seen after etching the pellet microstructure (Figure 9). A comparison of the microstructure of pellets etched for the same time shows poor granule-to-granule bonding in the pellet made from granules heat-treated at 1250°C. The etch had less effect

on the microstructure of the pellet made from granules heat treated at 1150°C. Thus, the higher the granule heat-treatment temperature, the lower the bond strength between granules.

Final Heat Treatment

Some effects of the final heat treatment can be observed in Figures 10 and 11. The as-pressed microstructure has considerable suboxide, while none is observed after a 1440°C heat treatment for 12 hr. In Figure 11, the grain size is about 10 μm after 12 hr at 1440°C, as compared to 3 to 4 μm after 6 hr at 1440°C.

Milliwatt Fuel Form Process

Critical Specifications

The most critical product specifications for Milliwatt Fuel are:

1. Minimum power density of 2.22 watts (thermal)/cc when mixed with yttrium.
2. Minimum amount of potentially respirable fines.
3. Chemical compatibility with tantalum alloy T-111 liner (not covered in this paper).

Development of Granule Process

Three potential flowsheets were investigated and are shown in Figure 12. It was found that presintering of the pellets before breakup produces strong granules with smooth cleavage faces and a minimum of asperities (surface irregularities), but decreases packing density. The first flowsheet investigated produced granules with the desired density, particle size, and grain size; however,

an undesirably high percentage of granules of less than 53 μm were present which required recycling (Table 1). The large amount of surface asperities present would produce an unacceptably high respirable fines content. This flowsheet was similar to the MHW granule process.

Flowsheet I was modified to omit the sieving of the less than 53 μm portion prior to presintering in an effort to reduce the percentage of once-through recycle (Flowsheet II, Table 1). Although the amount of recycle was reduced about 50% (Table 1), the granules produced also had a large amount of surface asperities (Figures 13 and 14).

An attempt was made to minimize asperities and fines content by strengthening the cold-pressed pellet by presintering for 3 hr at 1175°C prior to breakup. This presintering produced pellets of approximately 80% TD which broke into very acicular granules. Strength was increased to the extent that there was no evidence of granule rounding caused by abrasion after breakup. The smooth cleavage faces reduced the asperities after the 1650°C sintering (Figures 13 and 14).

Respirable Fines Content

The fines measurement technique developed at Mound Laboratory, Monsanto Research Corporation (MRC) which involves wet-sieving of the material was used to analyze fuel made by Flowsheets II and III. The results are shown in Table 1. Samples from Flowsheet I were not analyzed because the fines measurement technique was

not available at the time the granules were fabricated. The fines from Flowsheets I and II were expected to be similar. The fines contents of the presintered granules (Flowsheet III) compare very favorably with the fines contents of granules produced at MRC from calcined plutonium hydroxide. Typical fines content of MRC Milliwatt granules is 20 to 40 μg/g of fines of 5 to 10 μm.

Packing Density

Granules made from any one of the three flowsheets in Table 1 may be loaded to the required power density of 2.22 watts/cc (∿5.5 g/cc). However, granules prepared by Flowsheet III require packing with an electrical vibrator, while granules produced with Flowsheets I or II require only hand-tapping.

Test loading of the Type II granules was not considered necessary because their tap density (7.0 g/cc) was higher than that of Type I granules which could be packed to an acceptable density of 6.58 g/cc (Figure 15). Type III granules had a relatively low tap density (5.42 g/cc) because of their irregular shape; the assistance of electrical vibrator was required to obtain the required packing density.

The final internal density of the granules is expected to be relatively insensitive to variations in the exterior characteristics of the powder (particle size, agglomeration, powder morphology, etc.) because of sintering for long times at high temperatures. This is shown by minor differences in grain size and internal density (Table 1 and Figure 16). To maximize the

internal density of the Milliwatt granules (and thereby maximize the loading density), the calcination temperature and temperature of all operations prior to presintering must be limited to the minimum practicable, i.e., the 730°C nominal furnace temperature currently used for calcining SRP plutonium oxalate. The temperature is necessarily limited because fabrication operations involving increased calcination temperatures and reheating of calcined plutonium oxalate have been shown to decrease sintered densities [4].

If batch-to-batch variations in powder sinterability have an effect on product quality, it will probably be on fines content and packing density. Both of these variables are dependent upon the strength of the presintered pellet, whose strength will vary with presintered density. The sensitivity of the process to these variables is not known at this time.

REFERENCES

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3. D. P. H. Hasselman, *Ceramic Bull. 49* (1970) 12.

4. P. E. Potter, *J. Nucl. Mater. 15*, (1965) 100.

TABLE 1. Summary of MILLIWATT Flowsheet Study

	Flowsheet I	*Flowsheet II*	*Flowsheet III*
Final Tap Density, g/cc	6.7	7.05	5.4
Granule Internal Density, % TD[a]	95	96	95
Average Grain Size, μm	29	33	27
Fines Content, μg/g			
<5 μm[b]	-	93	8.2
5 to 10 μm[c]	-	997	0.53
Number of Operations	9	8	9
Once-Through Recycle, %	22	10	3

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a. TD = theoretical density of a PuO_2 granule.

b. Weight of particles of less than 5 μm per gram of granules.

c. Weight of particles measuring between 5 and 10 μm per gram of granules.

Multihundred Watt (MHW) Process	*MILLIWATT Process*
Precipitate Pu Oxalate	Precipitate Pu Oxalate
Calcine Pu Oxalate at 735°C	Calcine Pu Oxalate at 735°C
$^{16}O_2$ Exchange: 750-900°C, 2-8 hr	$^{16}O_2$ Exchange: 700°C, 2-8 hr
Outgas at 1000°C for 1 hr	Ball Mill 8 hr to 2 μm mode size
Ball Mill 8 hr to 2 μm mode size	Cold Press at 58,000 psi
Cold Press at 58,000 psi	Presinter at 1100°C for 3 hr
Breakup/Size <125 μm	Breakup/Size: 53-500 μm
Presinter at 1000-1250°C for 3 hr	Sinter at 1650°C for 10 hr
Hot Press at 1530°C and 3000 psi for 20 min	Ultrasonic Wash
Heat Treat at 1440°C for 12 hr	Vibratory Load
Encapsulate in Iridium	Encapsulate in Tantalum

FIGURE 1. Process Flowsheets for Fabrication of Fuel Forms from Plutonium Oxalate

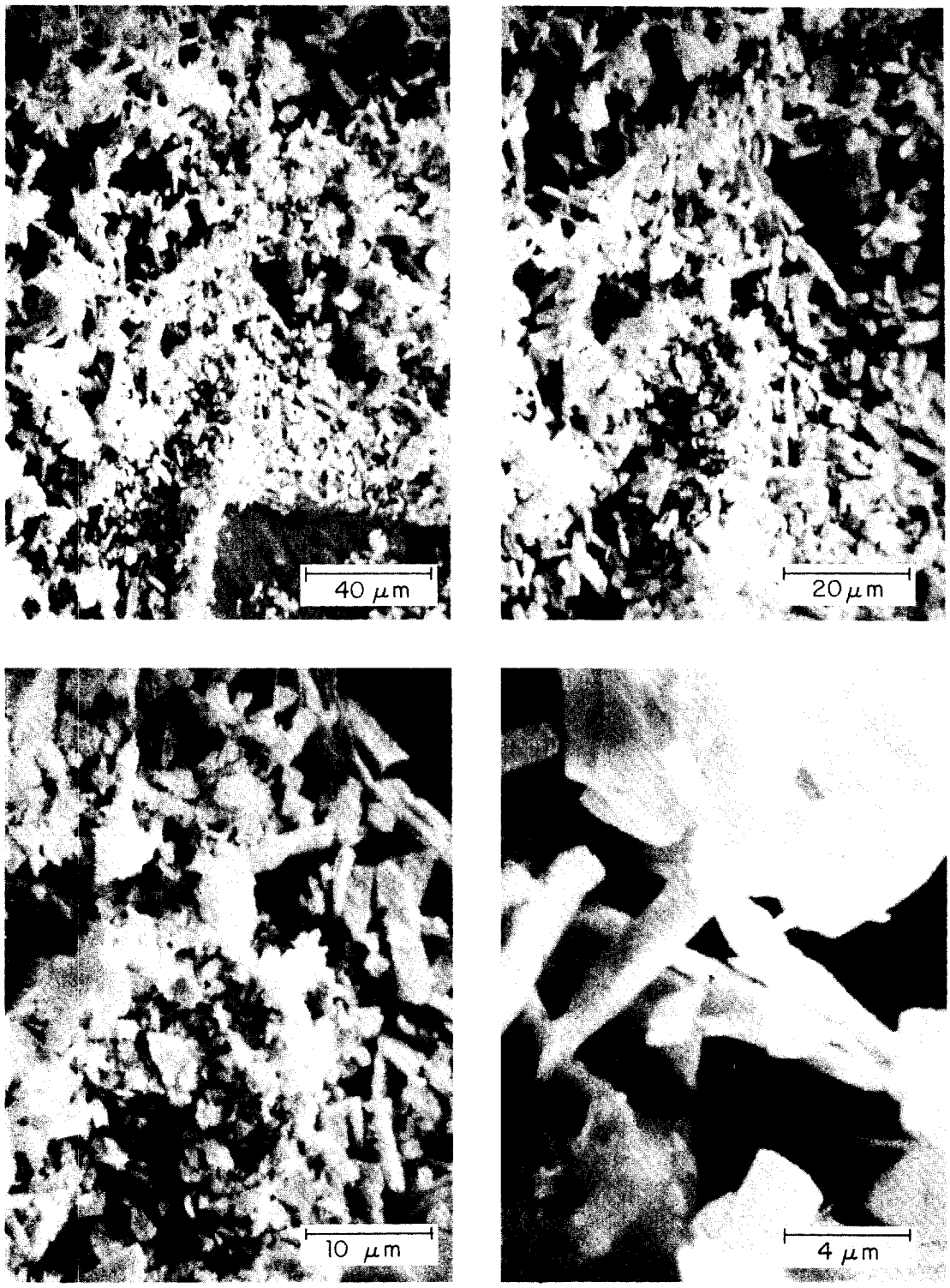


FIGURE 2. Particle Morphology of Calcined $^{238}PuO_2$ Powder Precipitated by the Present Reverse-Strike Production Technique

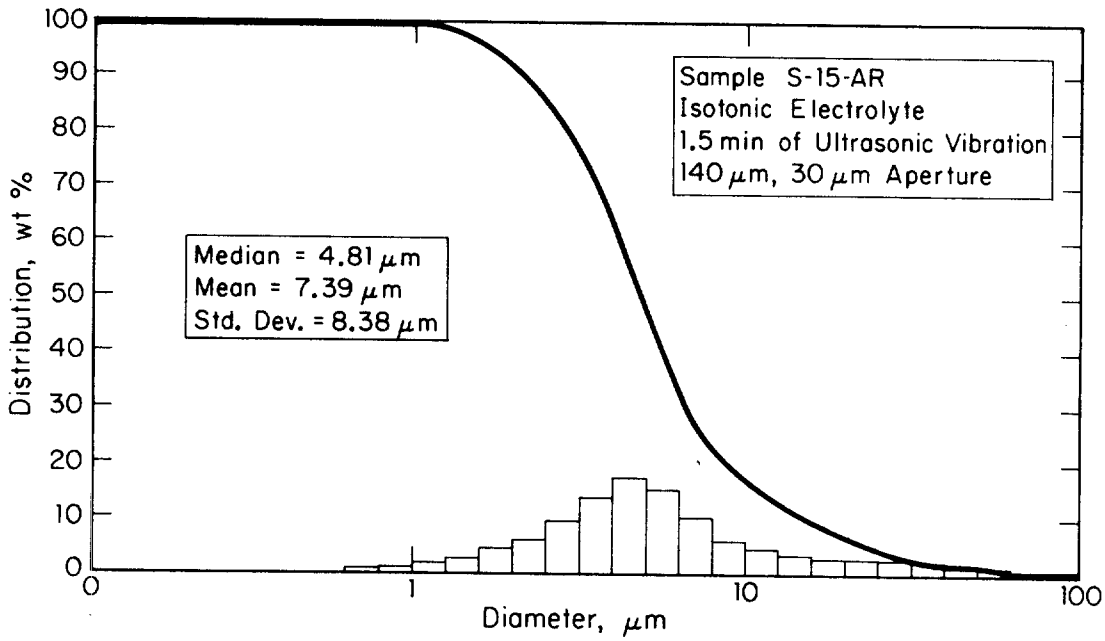


FIGURE 3. Particle Size Distribution of $^{238}PuO_2$ Powder

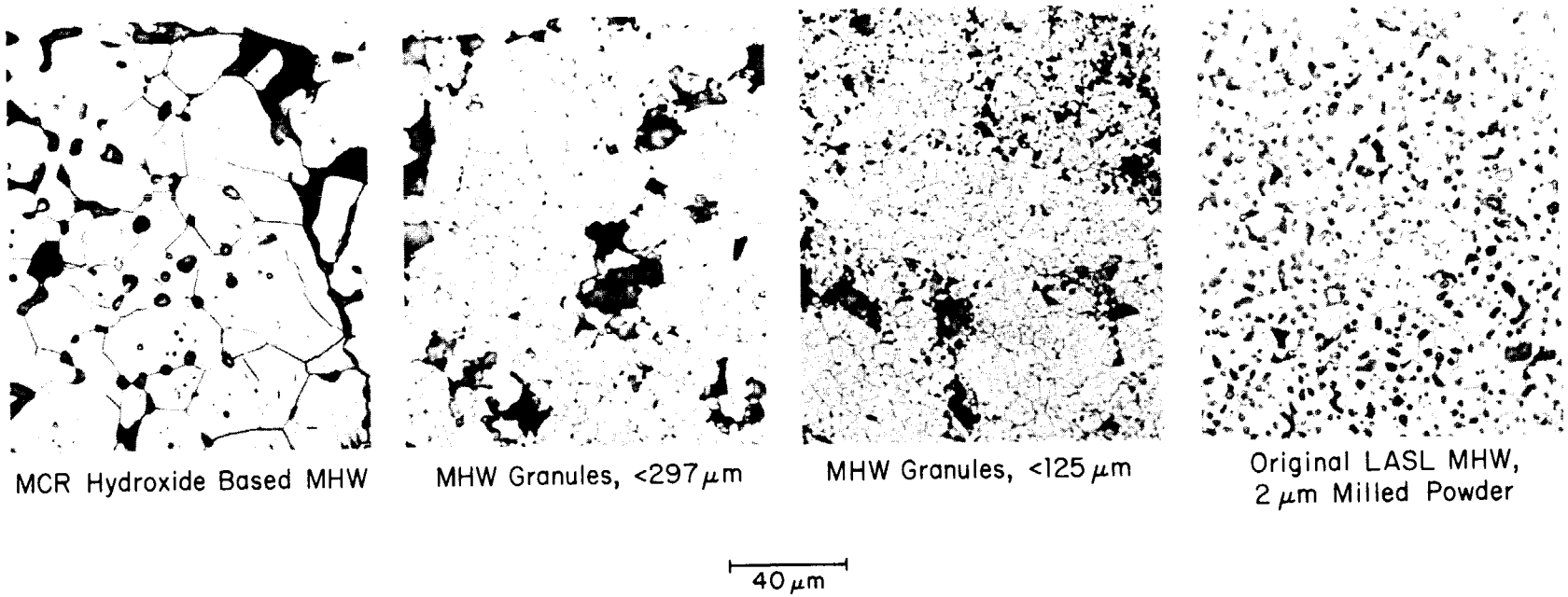
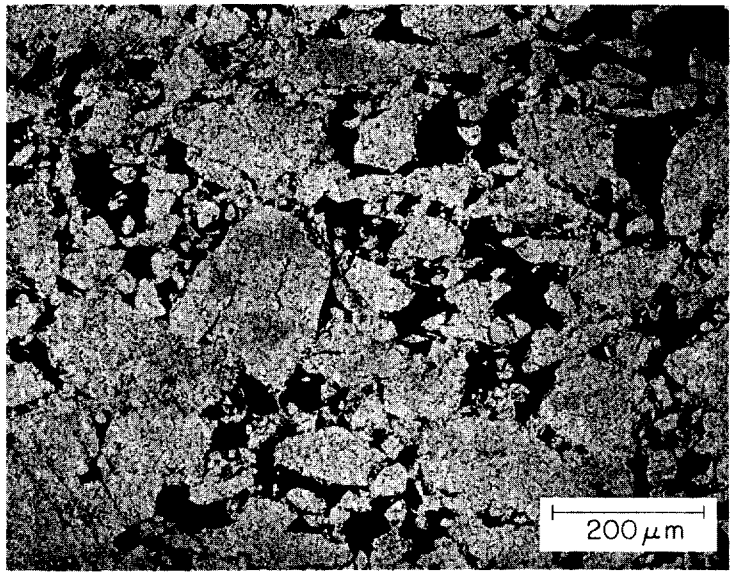
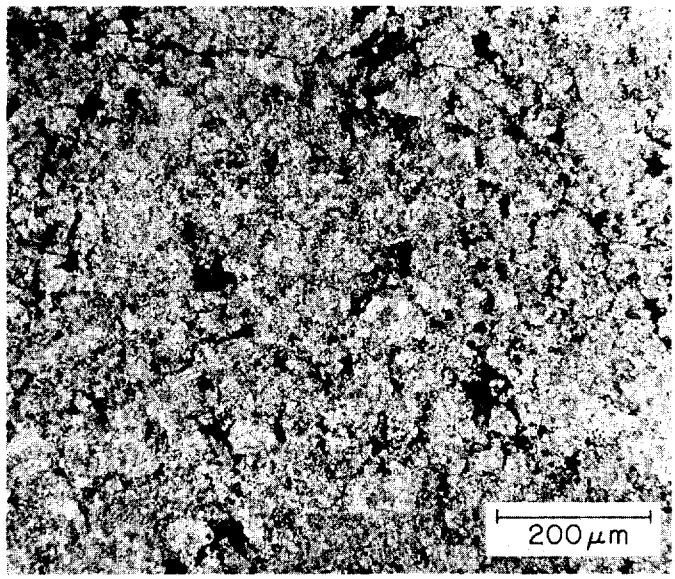


FIGURE 4. $^{238}PuO_2$ Heat Source Microstructures



Granules, <297 μm



Granules, <125 μm

FIGURE 5. Finer Feed Granules Result in More Uniform Microstructures

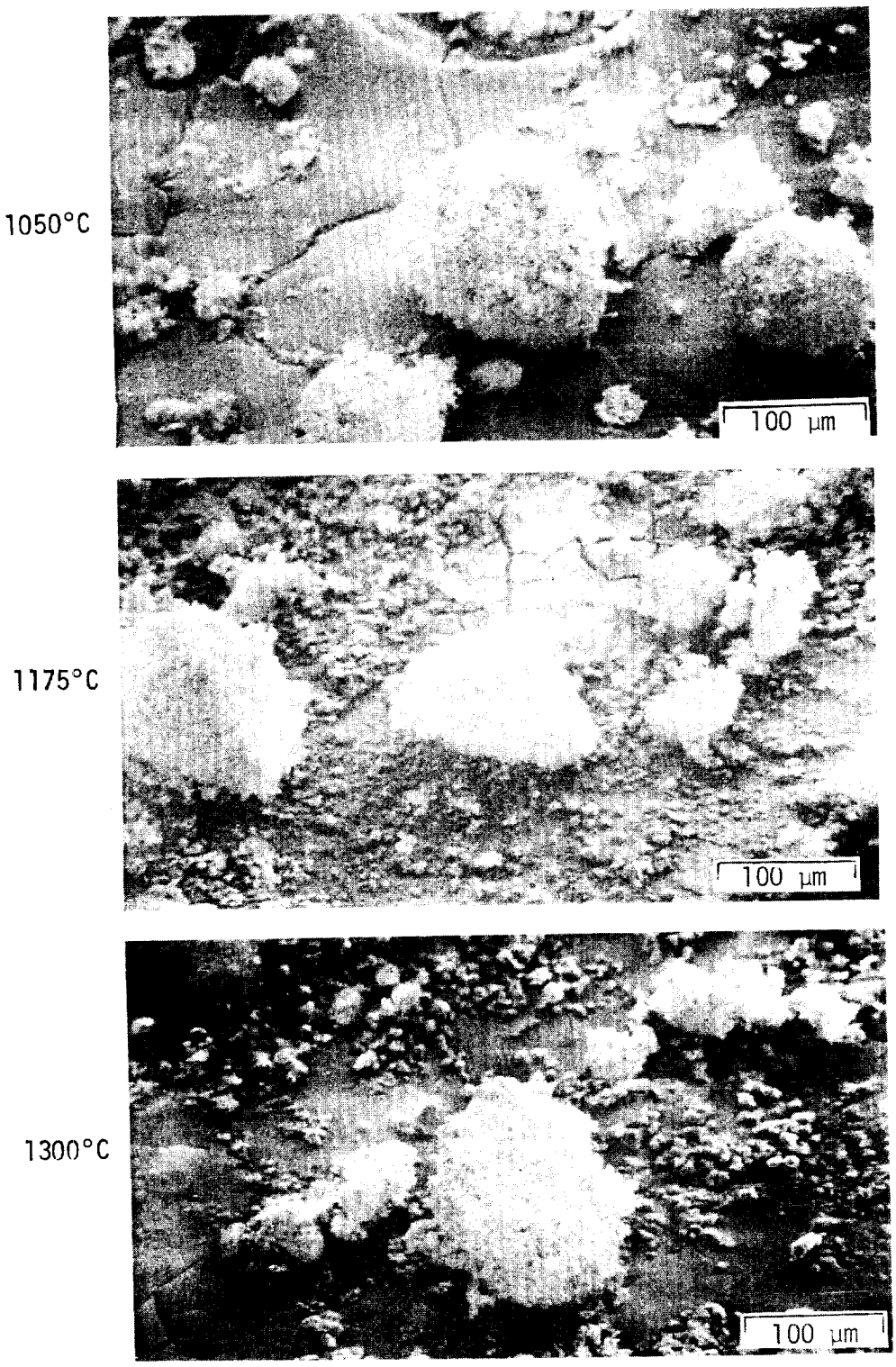
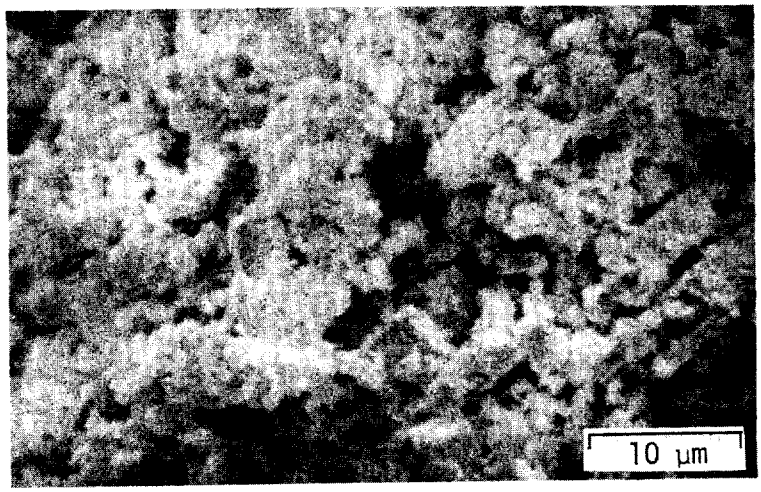
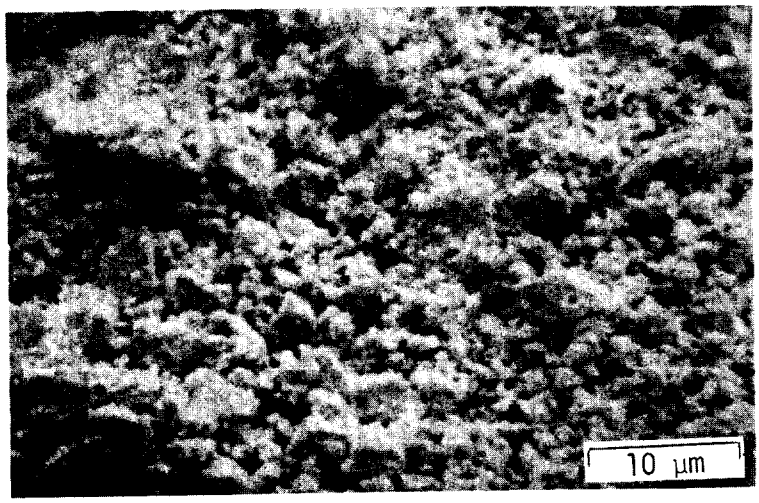


FIGURE 6. Scanning Electron Micrographs of Granules after 3-hour Heat Treatment

1050°C



1175°C



1300°C

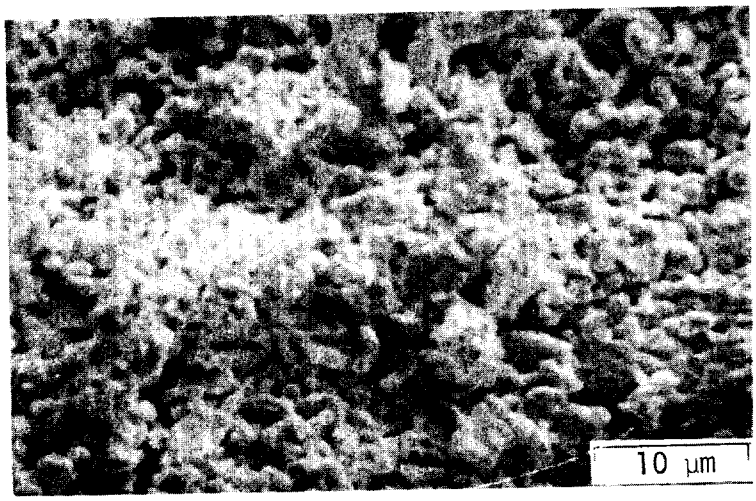
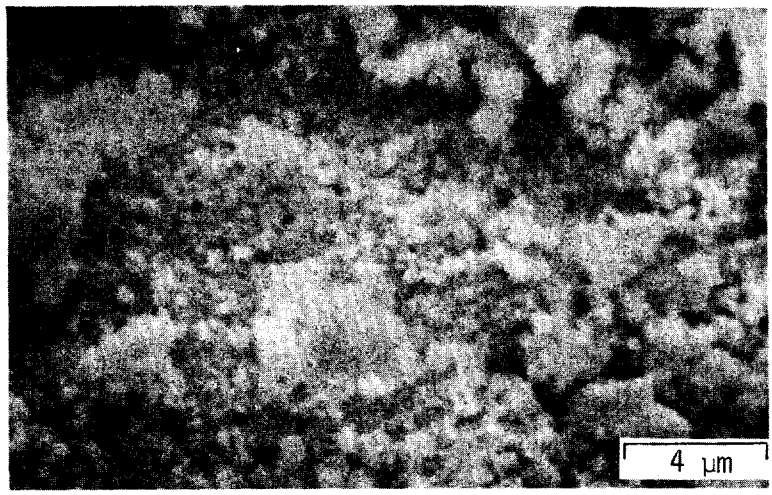
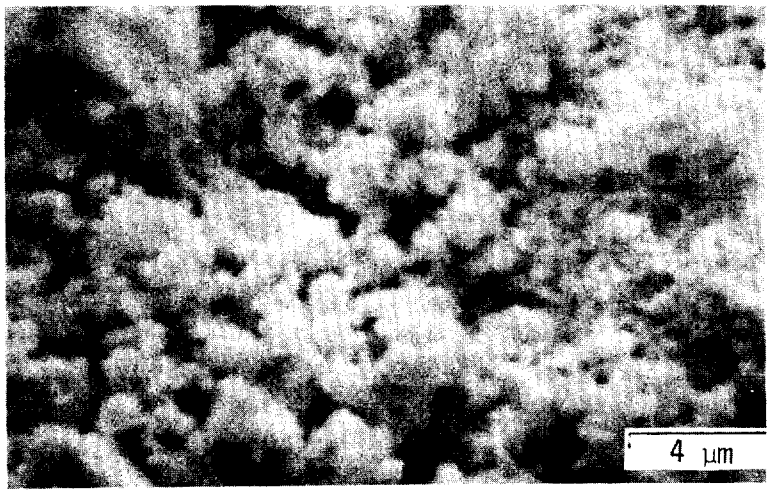


FIGURE 6 (Continued)

1050°C

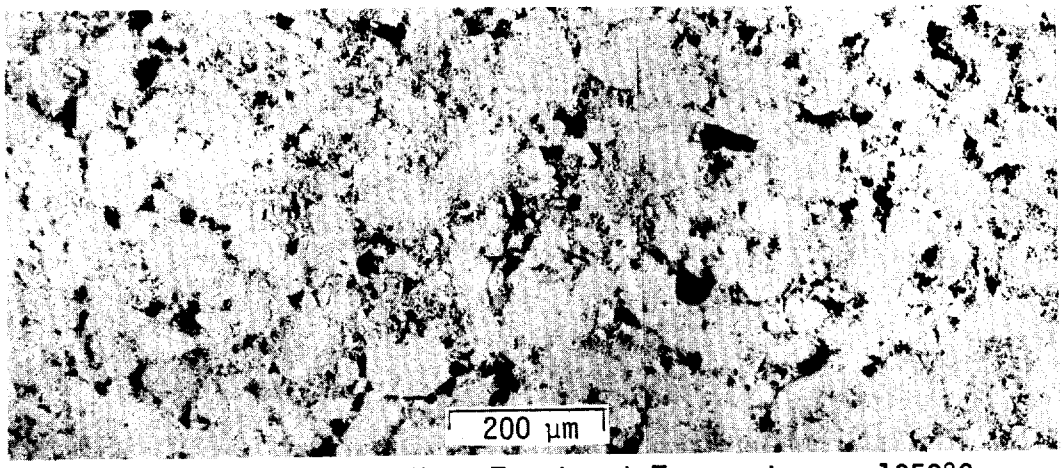


1175°C

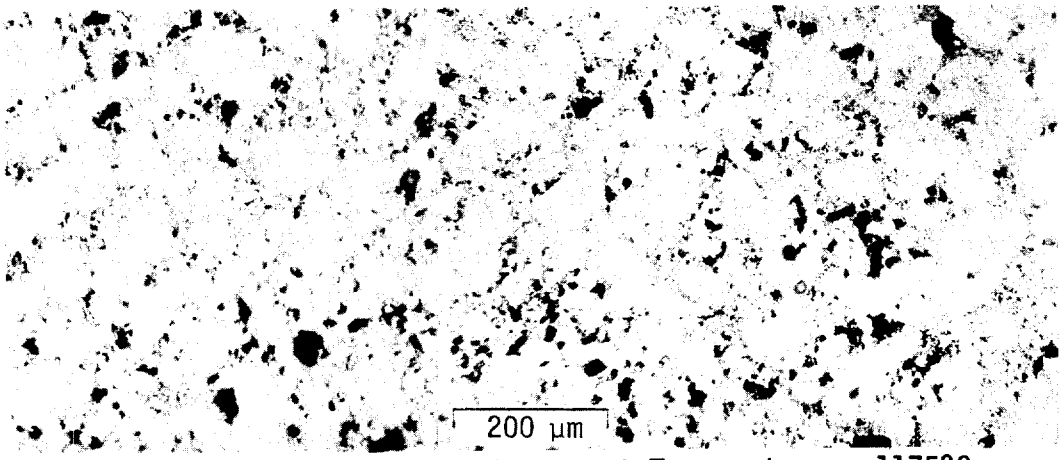


1300°C

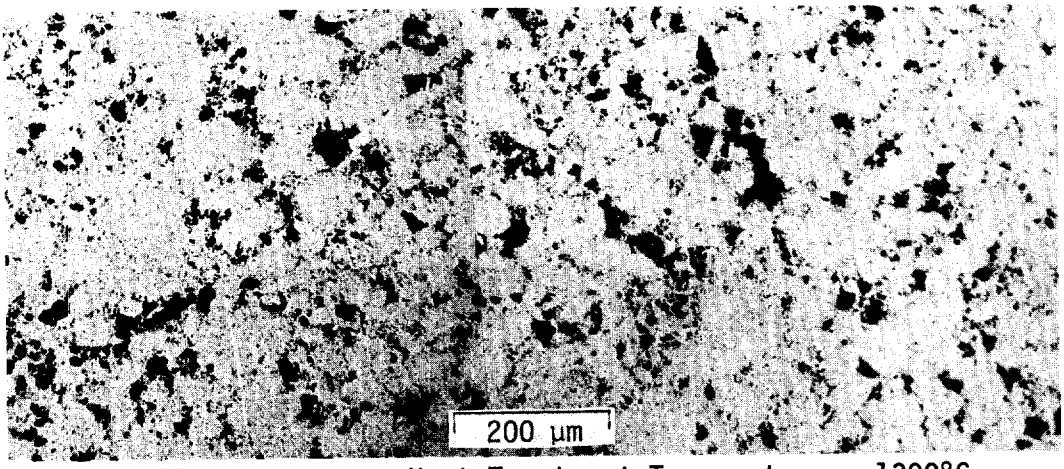
FIGURE 6 (Continued)



MHP-41 Heat Treatment Temperature: 1050°C



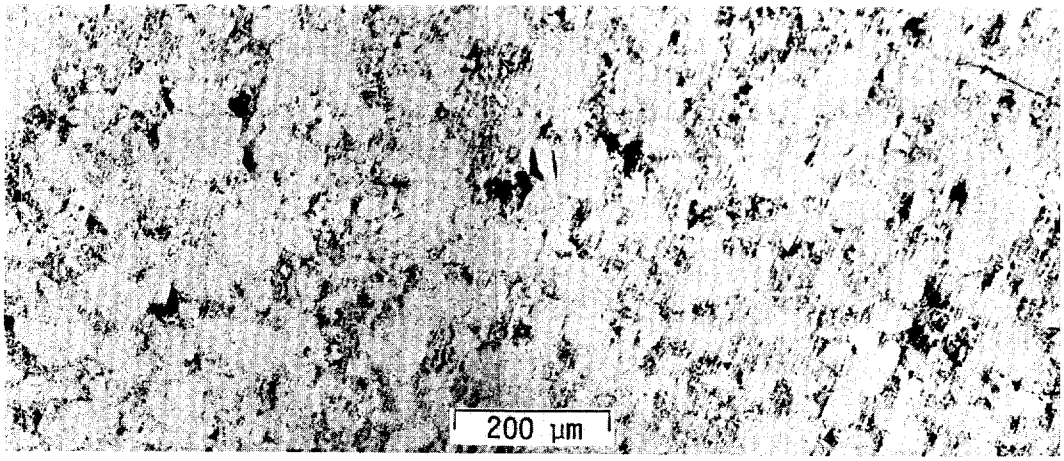
MHP-40 Heat Treatment Temperature: 1175°C



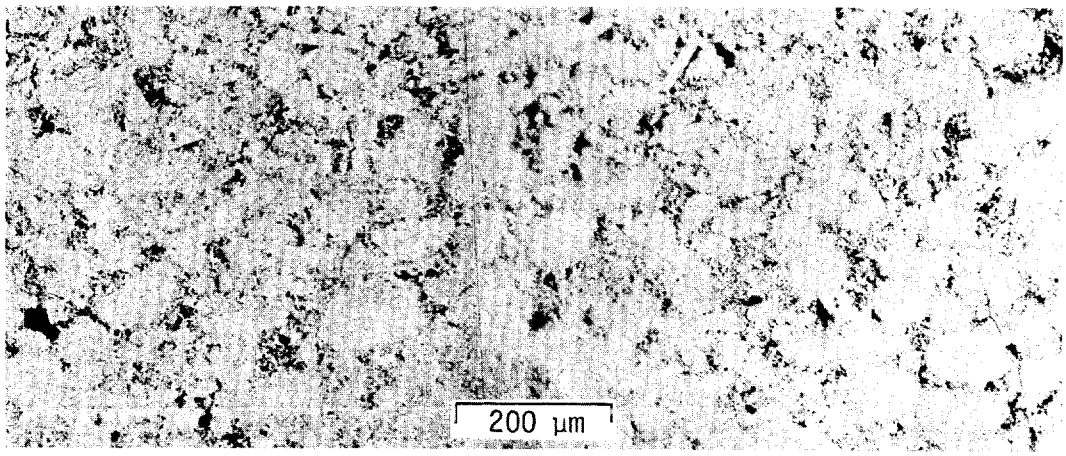
MHP-42 Heat Treatment Temperature: 1300°C

a. Hot Pressed at 3000 psi

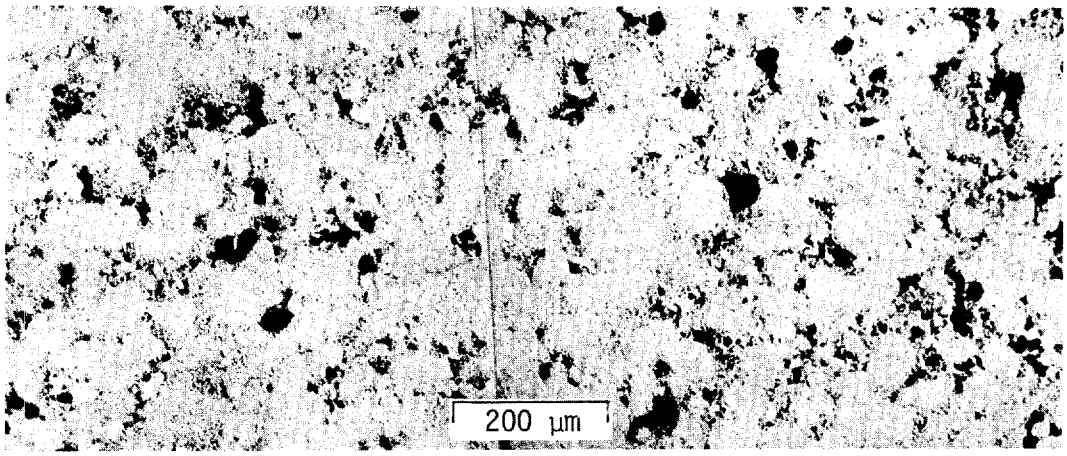
FIGURE 7. Effects of Granule 3-hour Heat-Treatment Temperature and Hot-Pressing Pressure on Pellet Microstructure



MHP-44 Heat Treatment Temperature: 1050°C



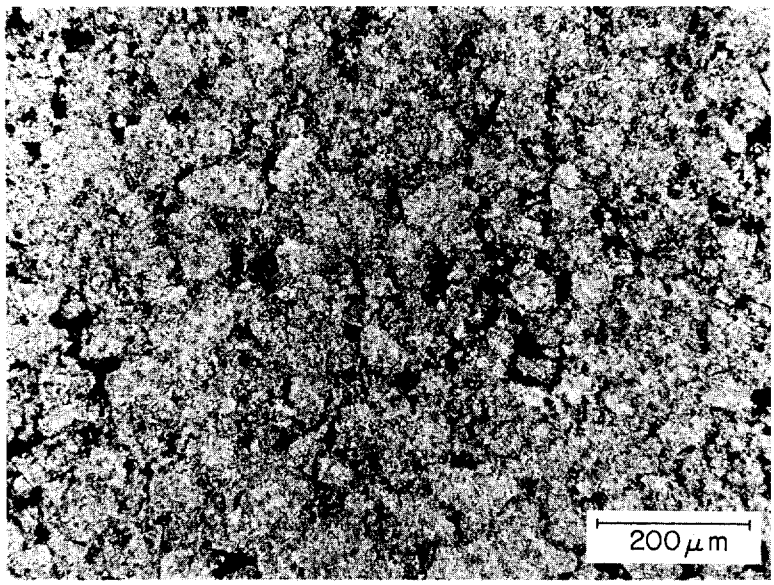
MHP-43 Heat Treatment Temperature: 1175°C



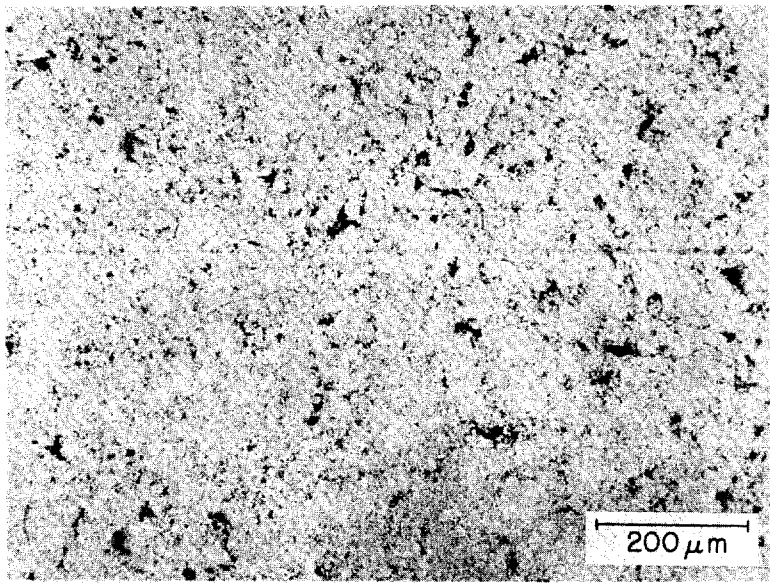
MHP-45 Heat Treatment Temperature: 1300°C

b. Hot Pressed at 5000 psi

FIGURE 7 (Continued)

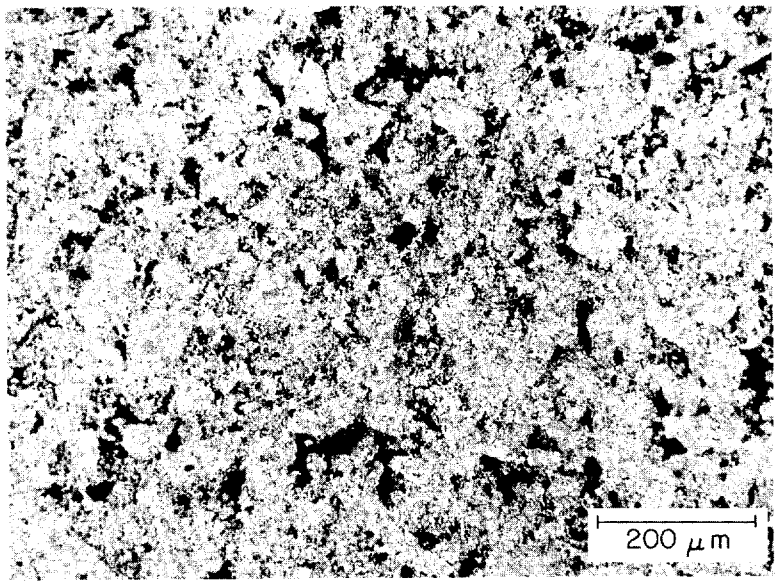


3000 psi, 83% dense

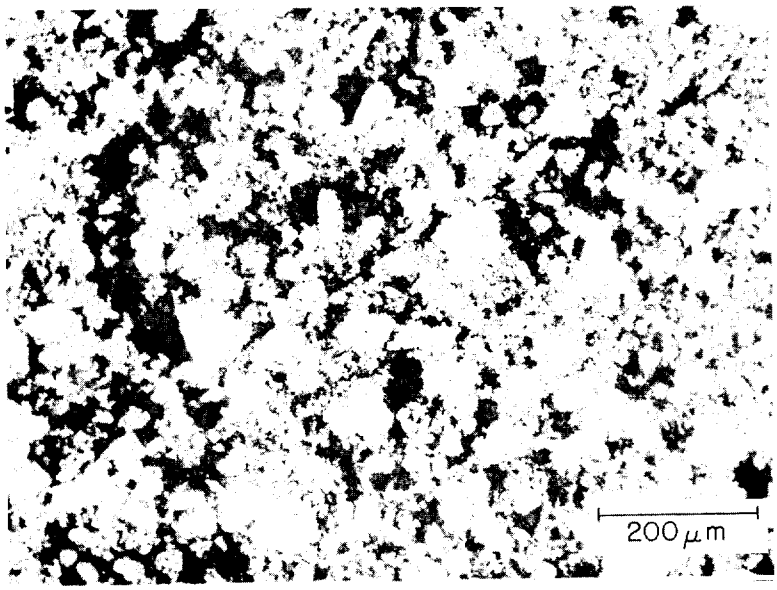


5000 psi, 90% dense

FIGURE 8. Effect of Hot-Pressing Pressure on Density and Microstructure

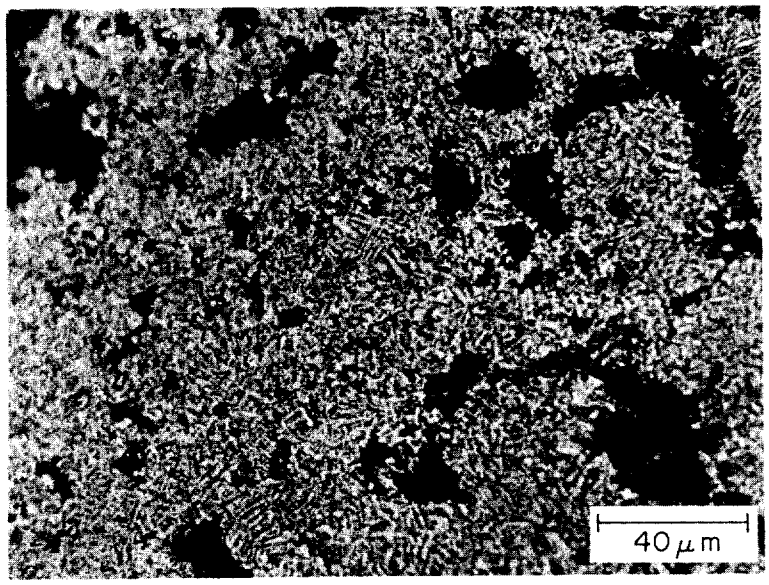


Etched pellet made from granules, 1150°C

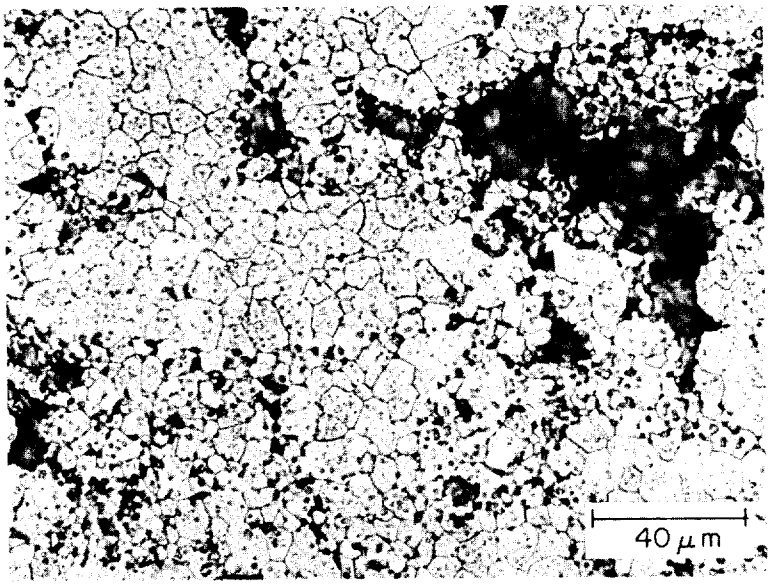


Etched pellet made from granules, 1250°C

FIGURE 9. Effect of Granule Heat-Treatment Temperature on Granule-to-Granule Bonding

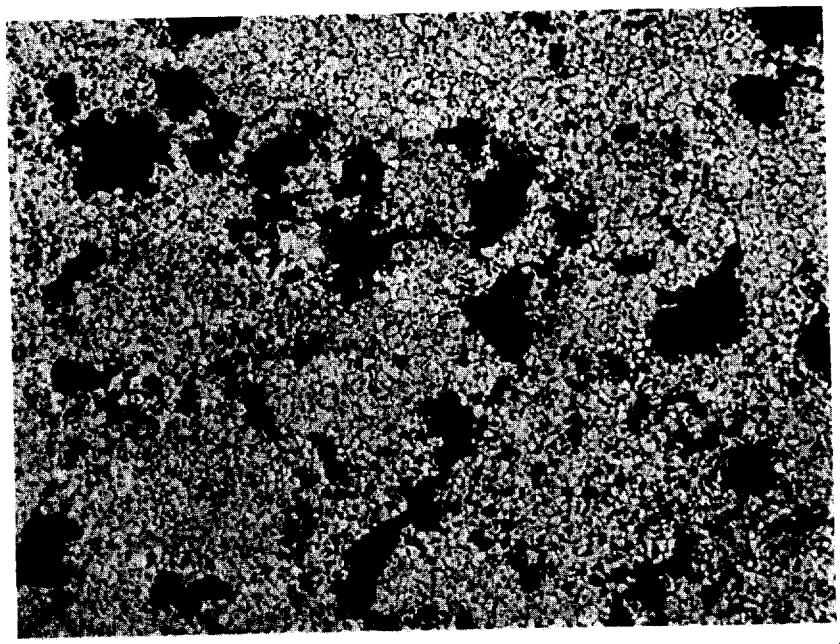


As-pressed



Heat treated at 1440°C for 12 hours

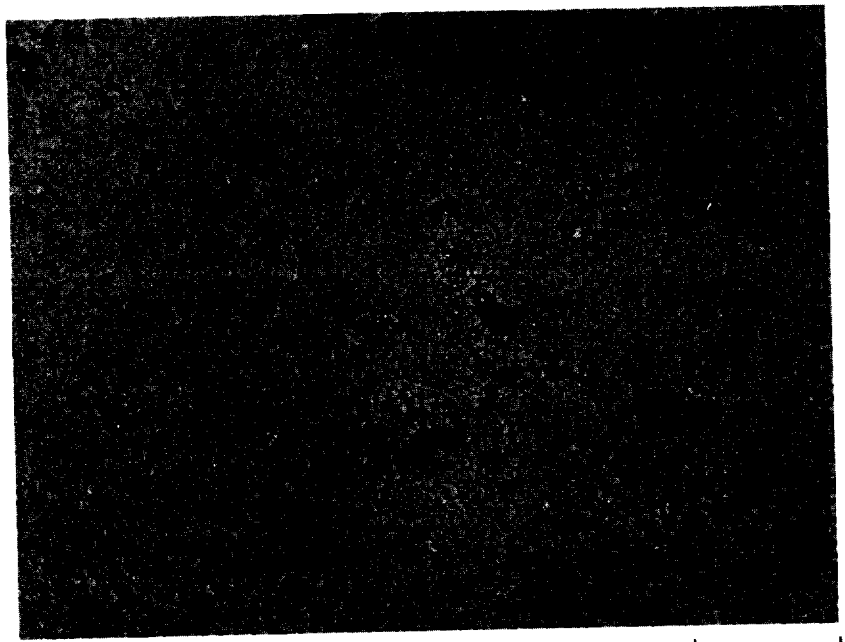
FIGURE 10. Granules with Reduced Coarse Porosity



Neg. No. 621-4

40 μm

6 hr (MHP-37-4)



Neg. No. 629-13

40 μm

12 hr (MHP-37-3)

FIGURE 11. Effect of Hold-Time at 1440°C on Grain Size

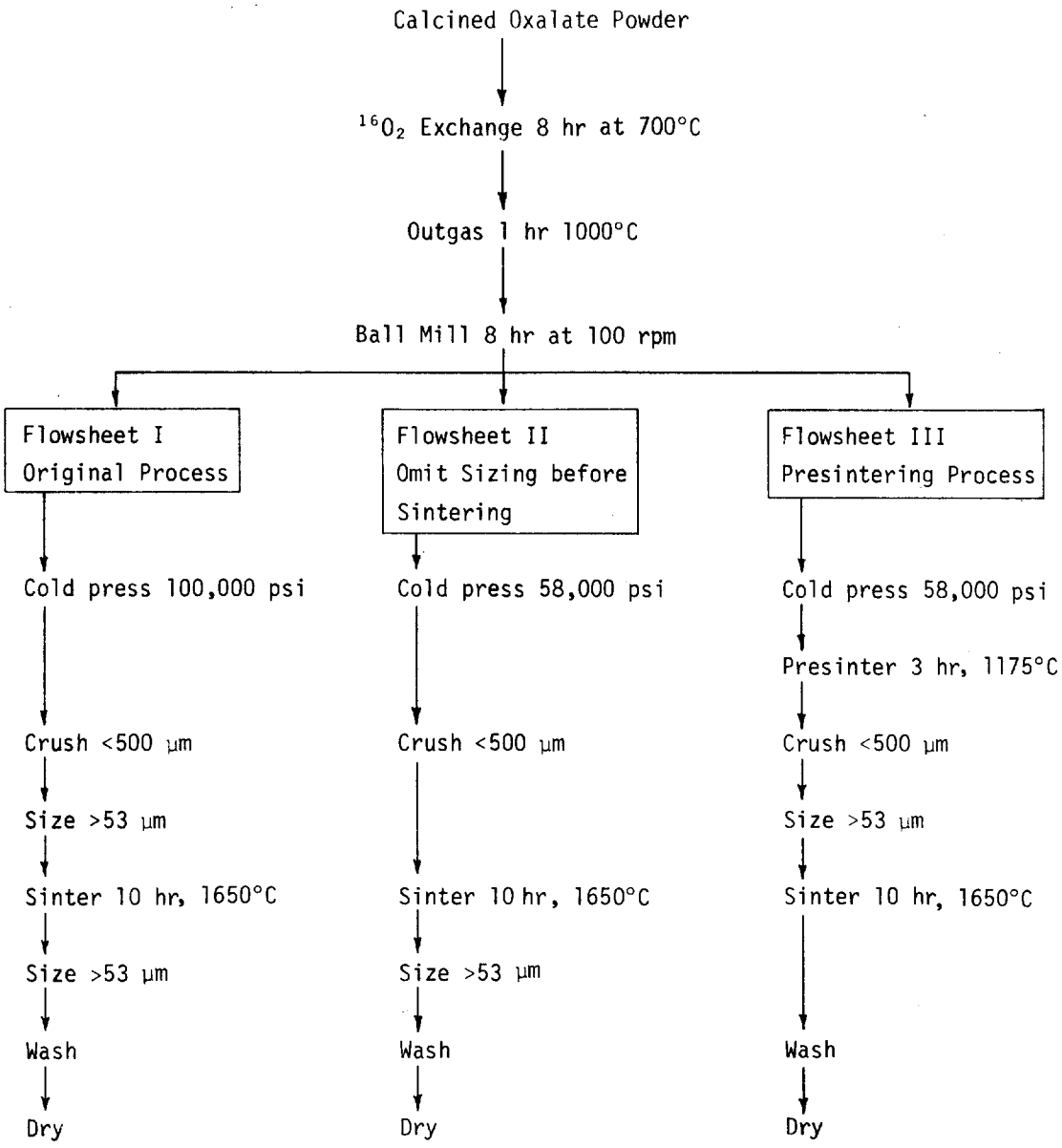


FIGURE 12. Milliwatt-RTG Granule Process Flowsheets

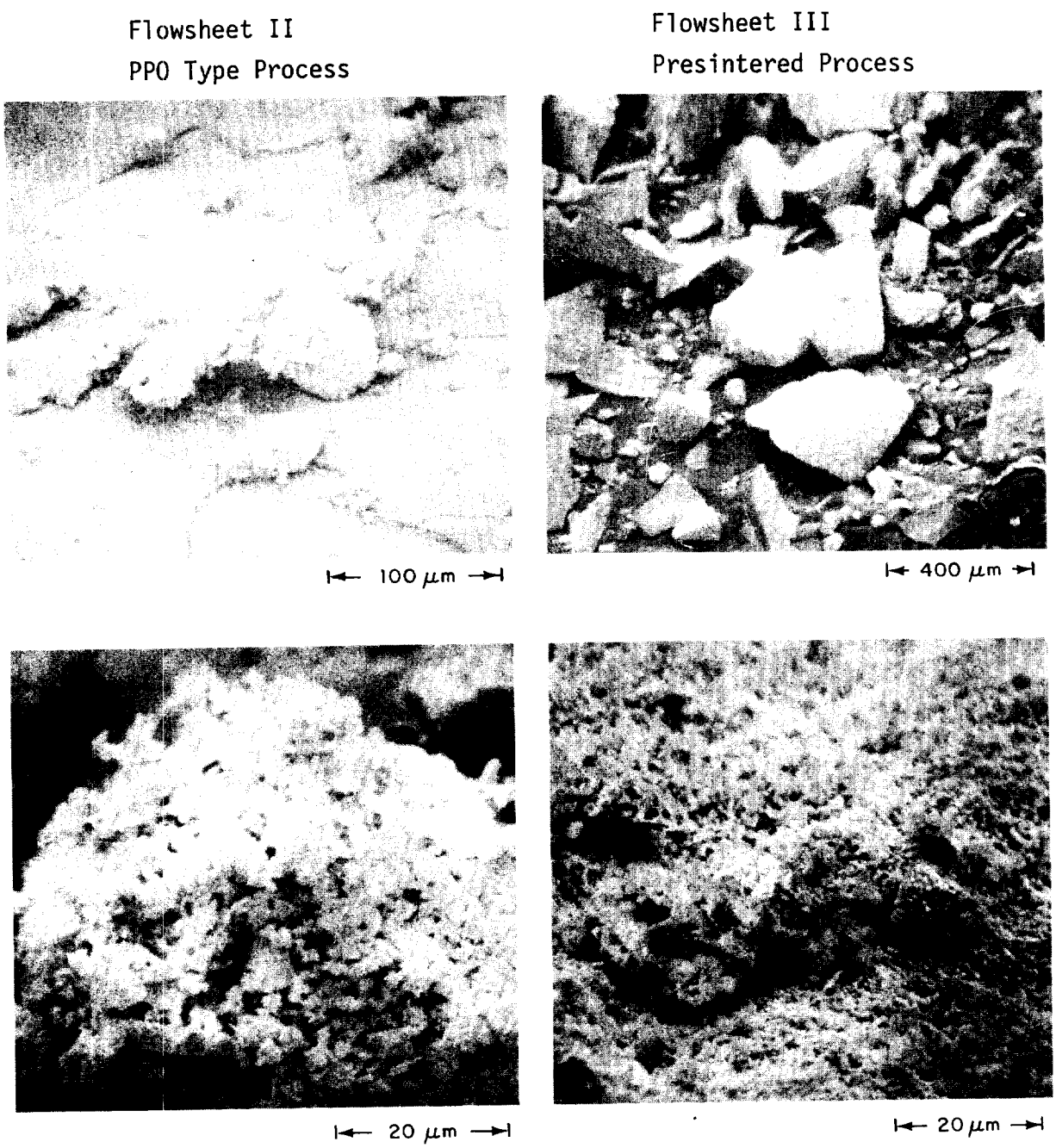


FIGURE 13. Structure of Milliwatt Granules before 1650°C Sintering

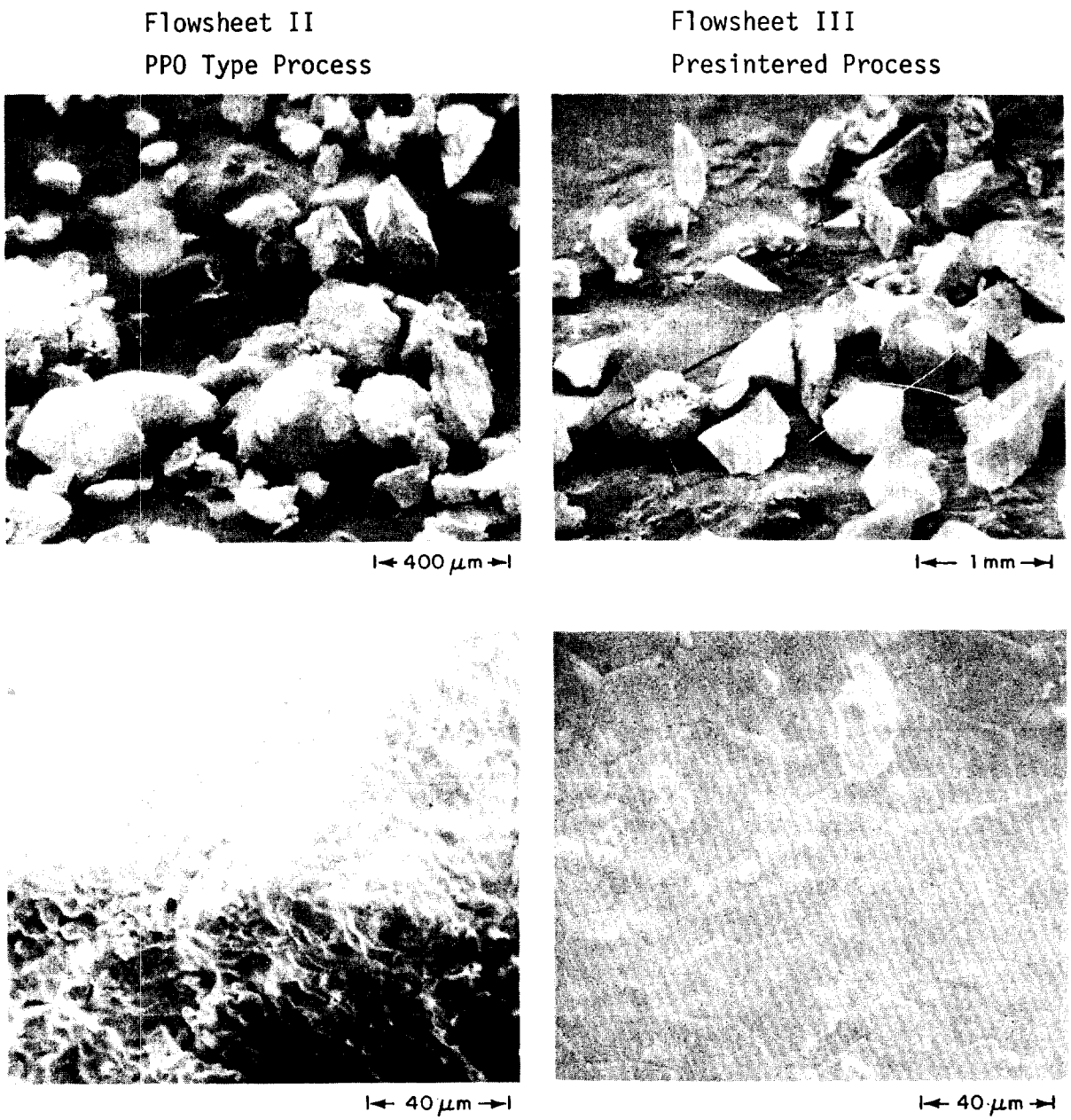


FIGURE 14. Structure of Milliwatt Granules after 1650°C Sintering

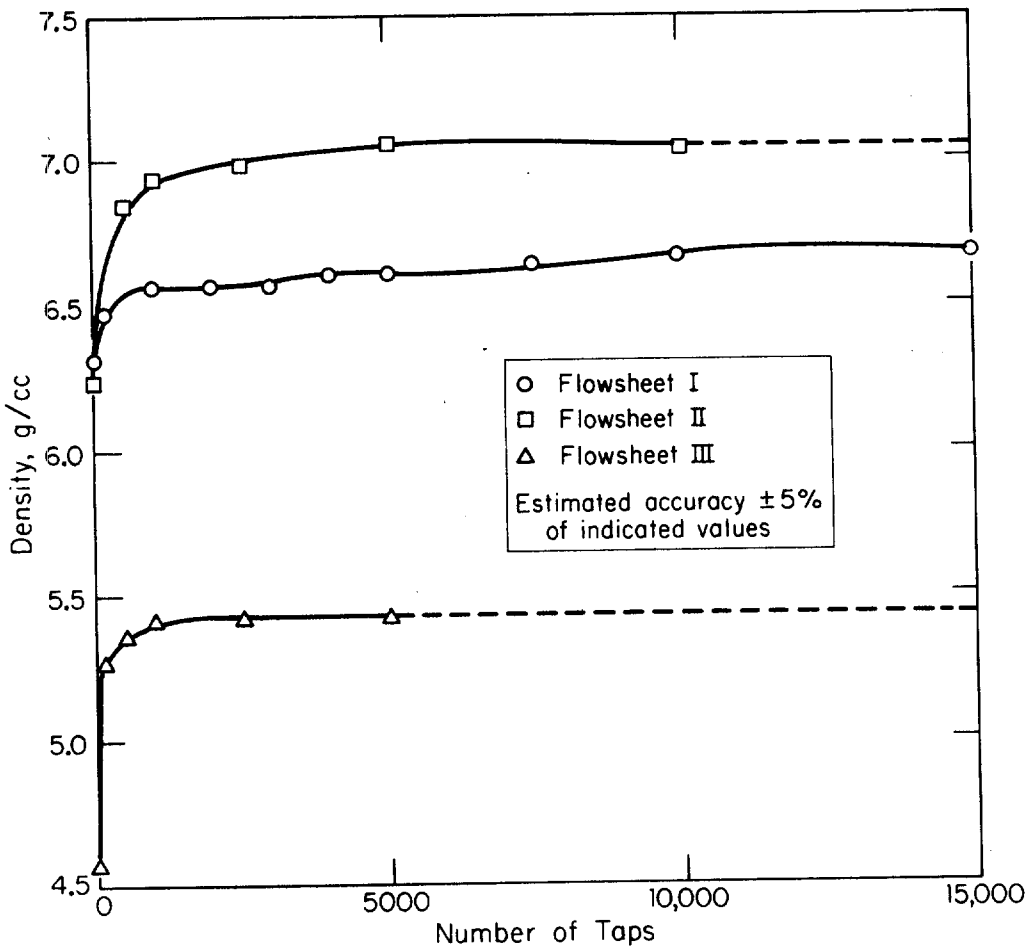
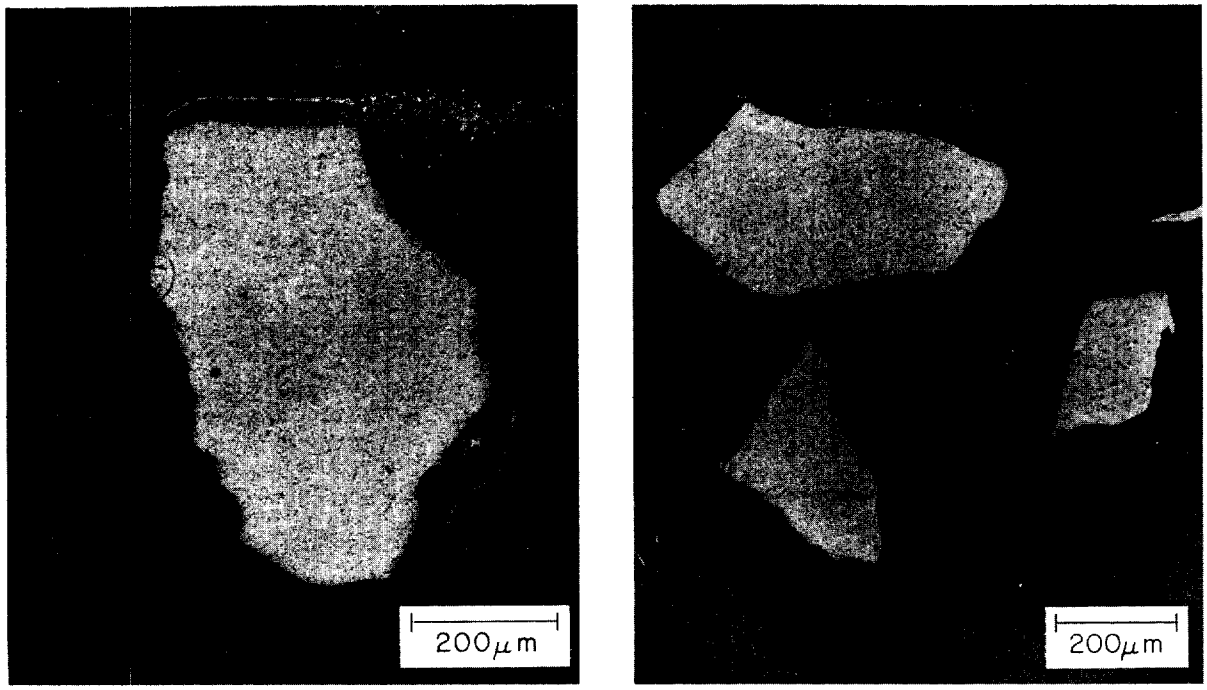


FIGURE 15. Tap Density of Granules Fabricated by Different Flowsheets

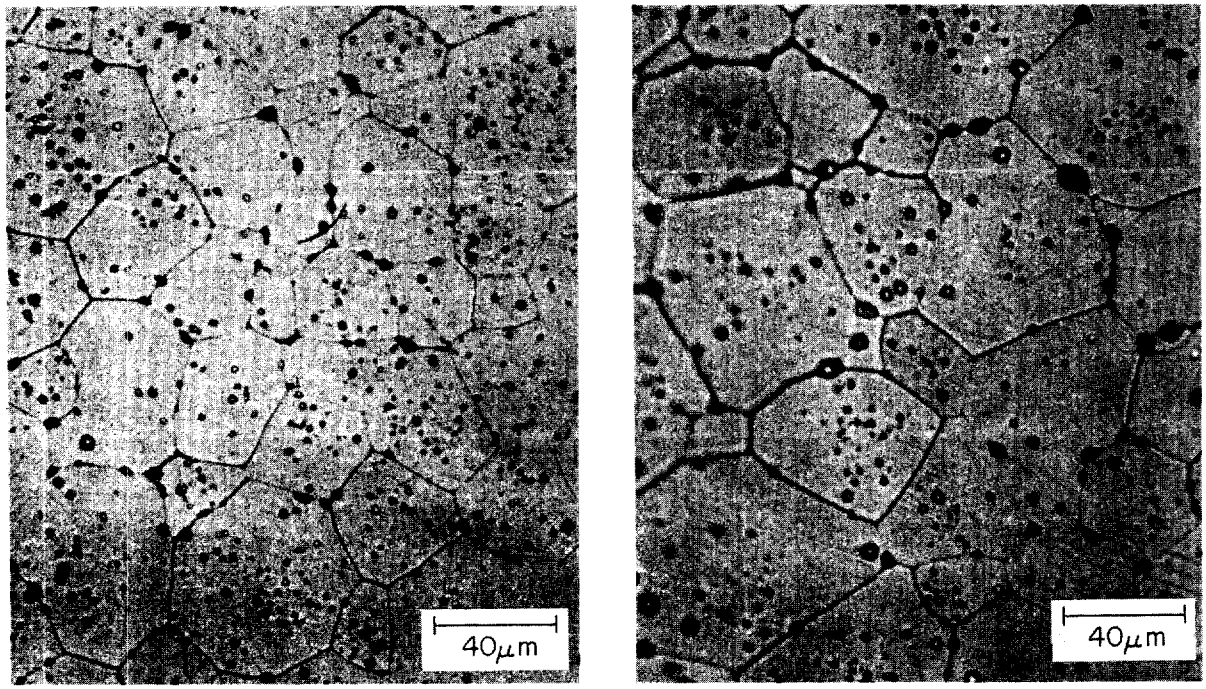
FLOWSHEET II

FLOWSHEET III



As Polished

As Polished



Etched

Etched

FIGURE 16. Typical Microstructure of Milliwatt Granules after 1650°C Sintering